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NEWS 3 MAY 08 CA/CAplus Indian patent publication number format defined
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NEWS 5 MAY 21 BIOSIS reloaded and enhanced with archival data
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NEWS 19 JUL 26 USPATFULL/USPAT2 enhanced with IPC reclassification
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NEWS 22 AUG 06 BEILSTEIN updated with new compounds
NEWS 23 AUG 06 FSTA enhanced with new thesaurus edition
NEWS 24 AUG 13 CA/CAplus enhanced with additional kind codes for granted patents
NEWS 25 AUG 20 CA/CAplus enhanced with CAS indexing in pre-1907 records
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NEWS EXPRESS 29 JUNE 2007: CURRENT WINDOWS VERSION IS V8.2,
CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
AND CURRENT DISCOVER FILE IS DATED 05 JULY 2007.

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| SINCE FILE
ENTRY | TOTAL
SESSION |
|---------------------|------------------|
| 0.21 | 0.21 |

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FILE LAST UPDATED: 27 Aug 2007 (20070827/ED)

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=> s olefin and distill
=> s olefin and distillation
 105038 OLEFIN
 103696 OLEFINS
 159968 OLEFIN
 (OLEFIN OR OLEFINS)
 58989 DISTILLATION
 422 DISTILLATIONS
 59144 DISTILLATION
 (DISTILLATION OR DISTILLATIONS)
 178561 DISTN
 1798 DISTNS
 179306 DISTN
 (DISTN OR DISTNS)
 197143 DISTILLATION
 (DISTILLATION OR DISTN)
 4941 OLEFIN AND DISTILLATION

=> propane and (propene or propylene) near separation
PROPANE IS NOT A RECOGNIZED COMMAND

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=> s propane and (propene or propylene) near sepatation
MISSING OPERATOR ROPYLENE) NEAR
The search profile that was entered contains terms or
nested terms that are not separated by a logical operator.

=> propane and (propene or propylene)

PROPANE IS NOT A RECOGNIZED COMMAND

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"HELP COMMANDS" at an arrow prompt (=>).

=> s propane and (propene or propylene)

83136 PROPANE

1129 PROPANES

83715 PROPANE

(PROPANE OR PROPANES)

75360 PROPENE

775 PROPENES

75697 PROPENE

(PROPENE OR PROPENES)

190060 PROPYLENE

305 PROPYLENES

190159 PROPYLENE

(PROPYLENE OR PROPYLENES)

L2 14686 PROPANE AND (PROPENE OR PROPYLENE)

=> s l1 and propane and (propene or propylene)

83136 PROPANE

1129 PROPANES

83715 PROPANE

(PROPANE OR PROPANES)

75360 PROPENE

775 PROPENES

75697 PROPENE

(PROPENE OR PROPENES)

190060 PROPYLENE

305 PROPYLENES

190159 PROPYLENE

(PROPYLENE OR PROPYLENES)

L3 207 L1 AND PROPANE AND (PROPENE OR PROPYLENE)

=> s l3 and (epoxidation or epoxide)

14673 EPOXIDATION

245 EPOXIDATIONS

14706 EPOXIDATION

(EPOXIDATION OR EPOXIDATIONS)

26255 EPOXIDN

570 EPOXIDNS

26343 EPOXIDN

(EPOXIDN OR EPOXIDNS)

28048 EPOXIDATION

(EPOXIDATION OR EPOXIDN)

50076 EPOXIDE

28851 EPOXIDES

65150 EPOXIDE

(EPOXIDE OR EPOXIDES)

L4 10 L3 AND (EPOXIDATION OR EPOXIDE)

=> d 14 1-10 abs ibib

L4 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN
 AB Procedure, for the continuous recycling an off-gas flow containing an unreacted olefin resulting by oxidation of olefins with hydroperoxides, includes following steps (1) compaction and cooling the off-gas flow, (2) separation the olefin from the off-gas flow by distillation, and (3) epoxidation of the separated olefin with a hydroperoxide. The procedure is especially useful for epoxidation of propane to propene oxide, whereby the off-gas flow contains propane and propane.

ACCESSION NUMBER: 2004:367231 CAPLUS

DOCUMENT NUMBER: 140:357189

TITLE: Procedure for the continuous recycling an unreacted olefin resulting by oxidation of olefins with hydroperoxides by compaction and pressure distillation

PATENT ASSIGNEE(S): BASF AG, Germany

SOURCE: Ger. Offen., 6 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|--|------|----------|------------------|------------|
| DE 10249378 | A1 | 20040506 | DE 2002-10249378 | 20021023 |
| CA 2503449 | A1 | 20040506 | CA 2003-2503449 | 20031023 |
| WO 2004037802 | A1 | 20040506 | WO 2003-EPI1736 | 20031023 |
| W: AE, AG, AL, AM, AT, AU, A2, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, C2, DE, DK, DM, D2, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, K2, LC, LK, LR, LS, LT, LU, LV, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, T2, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GO, GW, ML, MR, NE, SN, TD, TG, AU 2003278127 | A1 | 20040513 | AU 2003-278127 | 20031023 |
| EP 1558596 | A1 | 20050803 | EP 2003-769439 | 20031023 |
| R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK, CN 1705652 | A | 20051207 | CN 2003-80101850 | 20031023 |
| MX 2005PA04183 | A | 20050608 | MX 2005-PA4183 | 20050420 |
| US 2006058539 | A1 | 20060316 | US 2005-532096 | 20050421 |
| IN 2005CN00702 | A | 20070629 | IN 2005-CN702 | 20050421 |
| PRIORITY APPLN. INFO.: | | | DE 2002-10249378 | A 20021023 |
| | | | WO 2003-EPI1736 | W 20031023 |

PRIORITY APPLN. INFO.: WO 2003-EPI1736 W 20031023

L4 ANSWER 2 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN

AB A continuous process for the epoxidation of olefins (e.g., methyloxirane from propylene) with hydrogen peroxide using a product-stream predistill. step and unit is described and a process flow diagram presented.

ACCESSION NUMBER: 2001:581493 CAPLUS

DOCUMENT NUMBER: 135:137842

TITLE: Process for the epoxidation of olefins using a product-stream predistillation step and unit

INVENTOR(S): Hofen, Willi; Thiele, Georg; Möller, Alexander

PATENT ASSIGNEE(S): Degussa A.-G., Germany

SOURCE: Eur. Pat. Appl., 10 pp.

CODEN: EPXXDM

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|---|------|----------|-----------------|------------|
| EP 1122248 | A1 | 20010808 | EP 2000-102544 | 20000207 |
| R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, CA 2399129 | A1 | 20010809 | CA 2001-2399129 | 20010203 |
| WO 2001057010 | A1 | 20010809 | WO 2001-EP1166 | 20010203 |
| W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, CJ, CG, CI, CM, GA, GN, GO, GW, ML, MR, NE, SN, TD, TG, BR 200108063 | A | 20021105 | BR 2001-8063 | 20010203 |
| EP 1254126 | A1 | 20021106 | EP 2001-911586 | 20010203 |
| EP 1254126 | BI | 20030702 | | |
| R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, AT 244231 | T | 20030715 | AT 2001-911586 | 20010203 |
| JP 2003521544 | T | 20030715 | JP 2001-556860 | 20010203 |
| ES 2202281 | T3 | 20040401 | ES 2001-1911586 | 20010203 |
| ZA 2002005200 | A | 20030929 | ZA 2002-5200 | 20020627 |
| NO 2002003553 | A | 20020725 | NO 2002-3553 | 20020725 |
| US 2003114694 | A1 | 20030619 | US 2002-203184 | 20021004 |
| US 6646141 | B2 | 20031111 | | |
| PRIORITY APPLN. INFO.: | | | EP 2000-102544 | A 20000207 |
| | | | WO 2001-EP1166 | W 20010203 |

REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE REFORMAT

L4 ANSWER 3 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN
 AB An olefin is treated with an epoxidizing agent to give a mixture of an epoxide and a carboxylic acid by-product which is separated by a double extraction procedure. The mixture is partitioned using a solvent

immiscible with H₂O, which dissolves the epoxide, and an aqueous solvent to dissolve and extract the acid. Thus, a mixture containing epoxide derivs. of C₁₄-1-alkenes 34.3, AcOH 19, and EtOAc 46.7% was passed at 1660 g/hr into the center of a 5 cm diameter York-Scheibel column with 20 stages, operating at 25°. Water (3.3 parts/1 part AcOH) was introduced at the top of the column and 930 g/hr pentane at the bottom. The nonaq. phase withdrawn at the top contained 89% EtOAc and 0.6% AcOH while the aqueous phase at the bottom contained 99.4% AcOH and 11%

EtOAc, which was separated by distillation. A similar process was used to sep. a mixture containing AcOH, propylene, propane, EtOAc, and propylene oxide.

ACCESSION NUMBER: 1970:3346 CAPLUS

DOCUMENT NUMBER: 72:3346

TITLE: Recovery of hydrophobic epoxide compounds

INVENTOR(S): Taylor, Wallace Edmondson; Sehnert, Merle F.

PATENT ASSIGNEE(S): Celanese Corp.

SOURCE: Fr., 7 pp.

CODEN: FRXXAK

DOCUMENT TYPE: Patent

LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|------|----------|-----------------|----------|
| FR 1551419 | FR | 19681227 | FR | 19680110 |
| DE 1643852 | DE | | | |
| GB 1186333 | GB | | | |
| US 3541114 | US | 19701117 | US | 19670113 |
| PRIORITY APPLN. INFO.: | | | US | 19670113 |

L4 ANSWER 4 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN

AB The title compds. were prepared in a 2-step conversion process of olefins which were gaseous under normal conditions with AcOOH (I). Thus, 1680 g. propylene (II) and 676 g. I (20% by weight solution in AcOOH) were allowed to react per hr. in a 2-step reaction. The components

were passed into a reactor 360 mm. long and 150 mm. in diameter. The conversion in the 1st step took place at 50° in 50 min. and the 2nd step at 100° in 10 min.; in both steps the pressure was 100 atmospheric. Unreacted II was recycled after condensation at 50°. The reaction product was passed to a distillation column and subjected to fractional distillation at atmospheric pressure. Propylene oxide (III), b. 33-5°, was removed at the top at 402 g./hr., 77.9% yield with respect to I supplied, and 77.1% with respect to the nonconverted

II. II conversion was 22.5% by weight and I conversion was 95% by weight. The use of Me₂CO resulted in 362 g. III/hr. (70.1% with respect to II and 73% with respect to I). II conversion was 21.4% by weight and the I conversion was

97% by weight. The following results were obtained with a U4A reactor: 358 g.

III/hr.; III yield with respect to I, 69.5%; III yield with respect to II, 78%; I conversion, 98% by weight; II conversion, 19.8% by weight

The amount of distillation residue was 0.3 kg./kg. III formed while 13% CO₂ was formed with respect to 1 C atom of I. Corrosion gave 0.0001% by weight FeCl₃

in the reaction mixture with respect to I solution (1.1 g./m²/day). The same experiment was carried out with 0.01% by weight Na₅P3O10 and 0.01% by weight

quinoline as stabilizers. The results were: 465 g. III/hr.; III yield with respect to I, 90.1%; with respect to II, 87.6%; I conversion 97% by weight; and II conversion 22.6% by weight Isobutylene (3420 g.) and 201 by weight

I solution in EtOAc (4940 g.) was stabilized with 0.01% by weight Na₅P3.O10 and 0.01% by weight quinoline. The mol. ratio isobutylene-I was 4.7:1; in the

1st step, the temperature was 30° and the time 30 min.; in the second step, temperature 70°, time 6 min. Pure isobutylene oxide was obtained (88.2% with respect to I supplied).

ACCESSION NUMBER: 1966:473342 CAPLUS

DOCUMENT NUMBER: 65:73342

ORIGINAL REFERENCE NO.: 65:13655h,13656a-c

TITLE: Alkane epoxides

INVENTOR(S): Knapack, A.-G.

SOURCE: 11 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|------|----------|-----------------|----------|
| NL 6513640 | NL | 19660425 | NL 1965-13640 | 19651021 |
| BE 671268 | BE | | | |
| PRIORITY APPLN. INFO.: | | | DE | 19641022 |

L4 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
 AB cf. CA 57, 13716g. The (tert-Bu)2O₂ (I)-induced free radical addition of propylene oxide (II), styrene oxide (III), 1,2-epoxybutane (IV), 1,2-epoxyoctane (V), and 1,2-epoxy-3-phenoxypropene (VI) to 1-octene (VII) was studied in the temperature range 125–200°. A mixture of isomeric epoxybutanes distilled gave 2,3epoxybutane, b. 56–7°, n_D20 1.3815, and IV, b. 58.5–9.0°, n_D20 1.3832. The base-catalyzed reaction of H₂O with mesityl oxide according to Payne (CA 52, 16357f) gave 4-methyl-3,4-epoxy-2-pantanone (VIII), b₂₀ 61–2°, n_D20 1.4235. V (2 moles), 0.40 mole VII, and 0.06 mole I desorbed with N and heated 3 hrs. at 145°, the mixture freed from peroxide decomposition products and unreacted V and VII by distillation, the residue distilled at 0.10 mm. to remove the 1:1 adduct, and the product redistilled, gave 7.7% 7-hexadecanone, b₁₀ 82°, n_D20 1.4432, λ 5.85 μ failing to give pos. ketone test with Brady reagent and HONH₂.HCl, or to yield a semicarbazone.
 Gas-chromatographic comparison of its retention time with that of an authentic synthetic sample proved the assigned structure. IV (2 moles), 0.40 mole VII, and 0.06 mole I heated 3 hrs. at 145° in a desorated Parr autoclave yielded 3.43 g. 3-dodecanone, b₁₀ 15°, n_D20 1.4425, λ 5.85, 3.0. III (2 moles), 0.20 mole VII, and 0.03 mole I heated 3 hrs. at 150° under N and the mixture distilled gave 3.10 g. caprilenone (95% pure), b₁₀ 50 120–8°, n_D20 1.4975, 5.85. VI (1 mole), 0.10 mole VII, and 0.03 mole I heated 5 hrs. at 150° under N, the mixture distilled, and the solid (4.45 g., b₁₀ 170–200°) recrystd. 4 times from petr. ether gave a product, m. 83°, λ 5.85μ, mol. weight 527, consisting of 2:1 and 3:1 addition products (mol. weight 486 and 598). These results indicated that the chain transfer atom was the α-tertiary H atom on the epoxide ring.
 Formation of this product also indicated that the intermediate epoxy radicals, initially formed, rearranged to α-oxo radicals before addition to the olefin. A mechanistic path for this general reaction was outlined and discussed. II (3 moles), 0.20 mole VII, and 0.015 mole I in a desorated steel bomb heated 17 hrs. at 125° and the mixture distilled gave 2.33 g. 5-hydroxy-2-hexanone (IX), 80% pure, b₁₀ 60–90°, n_D20 1.4520, mol. weight 120.7, λ 3.0, 5.85 μ, together with 2.33 g. residue, mol. weight 640. II (2 moles), 0.20 mole VII, and 0.03 mole I heated 2 hrs. at 150° in a desorated Parr bomb and the mixture distilled left a residue of 11.2 g., n_D20 1.4595, mol. weight 324, and gave 5.34 g. product, b₁₀ 26 62–7°, n_D20 1.4368, mol. weight 211, λ 3.0, 5.85 μ, chromatographed to show the presence of 4.27 g. IX and 1.07 g. 2-undecanone (X). II (2 moles), 0.10 mole VII, and 0.12 mole I heated 1 hr. at 200° in a desorated Parr bomb and the mixture distilled left 10.2 g. residue, n_D20 1.4622, mol. weight 349, and gave 4.80 g. product, b₁₀ 26 50–7°, n_D20 1.4370, mol. weight 175.6, λ 3.0, 5.85 μ, containing 0.96 g. IX and 3.84 g. X; X semicarbazone m. 118–20°. With II, a secondary reaction, involving opening of the epoxide ring by intermediate acetylony radicals, produced IX. To ascertain the effect of ring size on the rate of H atom abstraction, the reactivities of the above epoxides II, III, IV, V, VI, VII as, well as trimethylene oxide, tetrahydrofuran, and tetrahydropyran toward Me₃CO₂ radicals were determined relative to C₆H₆. The reactant and I in 50:1 molar ratio was weighed into a glass ampul cooled in a solid CO₂-CHCl₃-CCL₄ bath, flushed with argon, the sealed ampul heated 40 hrs.

L4 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
 AB Olefins with more than 3 carbon atoms can be epoxidized in liquid phase by molecular oxygen using as catalyst olefinic aldehydes, that give the corresponding olefinic acids, and sulfur or aromatic nitro derivatives. Thus: A solution of 168 g. acrolein, 420 g. propylene, 180 g. nitrobenzene, and 1130 g. EtOAc is poured into a pressure vessel, heated at 100°, and saturated with oxygen for 1 hr. The distillation of the solution gives 397.2 g. unreacted propylene, 69.3 g. acrylic acid, (64%) 84 g. unreacted acrolein, and 28.4 g. 1,2-epoxypropane (90%). A solution of 112 g. acrolein, 420 g. propylene, 1369 g. EtOAc, and 5 g. sulfur is heated at 80°, and treated with molecular oxygen 2 hrs. to give (with recyclization) 82% 1,2-epoxypropane, and 70% acrylic acid; without sulfur the yields are 15% and 70%, respectively. A solution of 210 g. methacrolein, 420 g. propylene, 180 g. nitrobenzene, and 1130 g. EtOAc is heated at 95° and treated with oxygen for 16 min. to give (with recyclization) 85% 1,2-epoxypropane, and 86% methacrylic acid, (conversions 7.6% and 28%, respectively). A solution of 210 g. crotonaldehyde, 420 g. propylene, 180 g. nitrobenzene, and 1130 g. EtOAc is heated at 90° and treated with oxygen for 25 min. to give (with recyclization) 91% 1,2-epoxy-propane, and 65% crotonic acid (conversions 5.8%, and 66%, respectively).
 ACCESSION NUMBER: 1965:471400 CAPLUS
 DOCUMENT NUMBER: 63:71400
 ORIGINAL REFERENCE NO.: 63:13083f-h,13084a
 TITLE: Epoxidation of olefins in liquid phase with molecular oxygen
 INVENTOR(S): Lanos, Francoise; Clement, Genevieve
 PATENT ASSIGNEE(S): Institut Francais du Petrole, des Carburants et Lubrifiants
 SOURCE: 12 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|------|----------|-----------------|----------|
| FR 1401176 | --- | 19650521 | FR | 19630803 |
| PRIORITY APPLN. INFO.: | | | FR | 19630803 |

L4 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
 AB at 135 ± 1° (stirred oil bath), the cooled ampul opened, and the mixt. analyzed by gas chromatography. The reactivity studies were tabulated and results indicated that 5- and 6-membered cyclic ethers underwent H atom abstraction most readily. Trimethylene oxide, II, and IV were similar to C₆H₆ in reactivity, V and VI were intermediate in reactivity, whereas III, which contains a benzylic H, was the most reactive epoxide.
 ACCESSION NUMBER: 1963:421224 CAPLUS
 DOCUMENT NUMBER: 59:21224
 ORIGINAL REFERENCE NO.: 59:3748a,3749a-f
 TITLE: Free radical chemistry of cyclic ethers. IV. Free radical rearrangement of epoxides
 AUTHOR(S): Wallace, T. J.; Gratter, R. J.
 CORPORATE SOURCE: Univ. of Connecticut, Storrs
 SOURCE: Tetrahedron (1963), 19, 657-65
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable
 OTHER SOURCE(S): CASREACT 59:21224
 CODEN: TETRAB; ISSN: 0040-4020

L4 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN
 AB The autoxidn. of allyl chloride (II), methallyl chloride (III), 1,4-dichlorobutene-2 (III), and 3,4-dichlorobutene-1 (IV) formed chlorohydrins by attack at the double bonds. Analysis of the initial products from II implied an epoxide precursor was involved. Oxidation rates for III and IV were studied and show an unusual dependency on added anions. All allylic chlorides were fractionated. Oxidns. at atmospheric pressure were conducted using cylinder O. For pressure oxidns. an autoclave was used. Products from I and II were analyzed by gas chromatography. Without a catalyst the oxidation of II at 60° was very slow. A maximum oxidation rate of 5.5×10^{-7} mole O/mole olefin/sec. was reached in 12 hrs.; after 44.7 hrs. 0.064 mole of O/mole II was consumed, and the reaction terminated. In order to obtain a higher conversion and allow isolation of products 90.6 g. II containing 1.8 g. cobaltic acetyl-acetonate and 5 g. cumene hydroperoxide oxidized 46.3 hrs. at 60° and distillation of an 89-g. aliquot of the liquid product gave 40.7 g., b200 35-7°, 5.9 g., b200 78-9° (contained 1.6 milliequiv. of epoxide), 2.5 g., b50 55-75°, 4.5 g. b10 66-90°. At 200 lb./sq. in. 487.1 g. II containing 20 g. MgO and 4.8 g. tert-butyl hydroperoxide oxidized at 100° with an off gas, the reaction terminated at 375 min. after the consumption of 3.55 moles of O, the solid product separated, dissolved in H₂O, washed, acidified, and extracted with Et₂O gave 9.8 g. of liquid consisting mainly of HCO₂H. The 510 g. of liquid oxidation product gave a min. of 15 separate peaks on gas chromatography. Chloroacetone and 1,3-dichloro-2-methylpropanol-2 (V) constituted 24 and 11%, resp. Chloroacetone and 1,2-epoxy-3-chloro-2-methylpropane (VI) were identified by infrared gas spectra. V was purified by redistn., b14 62.8°, n_{20D} 1.4700, d₂₀ 1.250. The bis-2-naphthyl ether derivative m. 146-8°. Reaction with aqueous suspension of Ca(OH)₂ gave VII. Acidification of the salts yielded nearly all HCO₂H, a trace of AcOH, and no chloroacetic acid (43.2 min.). Attempts to titrate samples from various reaction stages for hydroperoxide iodometrically gave poor results. I (497.3 g.) containing 20 g. MgO at 100° and 200 lbs./sq. in. for 440 min. required 76.8 g. O and a constant maximum rate was reached in 260 min. ClCH₂CHO was identified. HCO₂H was present in the distillate. 1,3-Dichloropropanol was isolated as a pure liquid; phenylethane m. 172-3°. The acid components (8.5 g.) extracted from the acidified MgO contained equal amounts of chloroacetic acid and HCO₂H. The oxidation of III at 90° was terminated after the consumption of 0.74 mole O at 27 hrs. Several tenths of a gram of (CoCl₂H₂O) was directly filtered. The solution was found to contain 2.2 milliequiv./g. of acid. The solution, 81 g., taken up in CHCl₃, washed, dried, and distilled gave 13 g. unchanged III. No 1,3-isomer was obtained. Trichlorobutanol in 28% yield was collected at 68-77°/1 mm., n_{25D} 1.4980. The oxidation of 2M III in AcOH with 0.1M cobaltous chloride gave 0.19 mole HCl and 0.13

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 GI For diagram(s), see printed CA Issue.
 AB cf. C.A. 50, 24121. Me₂CHC(OH)Ph₂ (I) gave 60% Me₂C:CPH₂ (II) with Ac₂O and 50% with o-C₆H₄(CO)₂ (IIa); it was best prepared by keeping 40 g. I, 50°, C₅H₅N, and 42 g. POC₁₃ at room temperature several days, pouring on ice, extracting with Et₂O, drying the exts., concentrating, distilling, refluxing the distillate 6 hrs. with Na, and redistg. to give pure II, b1.6 114°, n_{23D} 1.586. II (20 g.) in 100 cc. Ac₂O treated in 0.5 hr., with cooling, with 15 g. Cr₂O₃ in 80 cc. Ac₂O, stirred 3 hrs., poured into 1.5 l. H₂O, and the product extracted with Et₂O gave 14.4 g. CMe₂CPH₂ (III), b0.7 102-12°, m. 64° (alc.); by-products of the reaction were identified as Ph₂CO and Me₂CO. II and Bz₂O₂ also gave III, b0.25 88°. III (0.2 g.) and 5 cc. H₂SO₄ shaken several days gave a diol, m. 94-5° (petr. ether). II reacted vigorously with Cr₂O₃ in 65% H₂SO₄, giving no III, but instead, Ph₂CO and Me₂CO. p-MeC₆H₄CH(OH)CHMe₂ and saturated Et₂O-HCl gave p-MeC₆H₄CHClCHMe₂ which, treated in situ, with C₅H₅N gave p-MeC₆H₄C(Me₂)₂ (IV), m. 58-9° (alc.). As above, 20 g. IV and 11 g. Cr₂O₃ in Ac₂O gave 13 g. epoxide (V), b1.5-1.7 139-42° [by-products were (p-MeC₆H₄)₂CO and Me₂CO]; IV and Bz₂O₂ also gave V, m. 58-9°. V, as above, gave a diol, identified by oxidation with HIO₄ to (p-MeC₆H₄)₂CO (VI). IV and Cr₂O₃ in 43% H₂SO₄ gave VII but no V. p-BrC₆H₄MgBr (from Bz₂O₂), q. p-BrC₆H₄Br and 60 g. Mg) and 110 g. Me₂CHCO₂Br gave crude (p-BrC₆H₄)₂C(OH)CHMe₂ (VII); distillation of VII gave (p-BrC₆H₄)₂C(Me₂)₂ (VIII), b0.7 174°, m. 97° (alc.); as above, 15 g. VIII gave 10 g. epoxide (IX), b1.2 177-8°, m. 94-5° (alc.); IX was unaffected by 43% H₂SO₄ at room temperature for 3 days. Similarly were prepared (p-ClC₆H₄)₂C(OH)CHMe₂, (p-ClC₆H₄)₂C(Me₂), b1.4023.

ACCESSION-NUMBER: 1958:25393 CAPLUS
 DOCUMENT NUMBER: 52:25393
 ORIGINAL REFERENCE NO.: 52:4561g-i,4562a-b
 TITLE: Reactions of unsaturated compounds. XIII. Oxidation of 1,1-diarylolefins by chromic oxide
 AUTHOR(S): Hickinbottom, W. J.; Moussa, G. E. M.
 CORPORATE SOURCE: Univ. London
 SOURCE: Journal of the Chemical Society (1957) 4195-8
 CODEN: JCSOA9; ISSN: 0368-1769
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

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 mole CO₂/mole of III. Dist. gave no sepn. of products. An 82% yield of a liquid b3 73-160° consisted of esters contaminated with acids, olefinic, and hydroxy compds. Titration of the crude oxdn. mixt. for hydroperoxide was unsuccessful. Oxidn. of IV at 90° with 0.5% Co naphthenate consumed 0.16 mole O in 24 hrs., and purification of the product gave 35 g. IV and 7.6 g. III. No isomer was recovered for reaction in the absence of catalyst. The combined products from oxdn. of 2M IV in AcOH representing 225 g. starting material were distd. The AcOH forerun of 29 g. yielded an unstable 2,4-dinitrophenylhydrazone, m. 125°. From the 18.5 g. fraction, b4 54-66°, 4.5 g. crystals were obtained, m. 62-3°, C₁₇H₂₀O₂. The p-bromophenacyl ester m. 97-9°. The ester fraction, b4 72-81°, 14.5 g., on hydrolysis with 1% H₂SO₄ and steam distn. gave AcOH and a liquid from which 1,2,3,4-tetra-chlorobutane was obtained as platelets, m. 73°. The remaining liquid was trichlorobutanol; urethan m. 132-3°. The next highest boiling distillate, b1 81-127°, 8.6 g., appeared to be a mixt. of esters. And the last fraction of 17 g., b1.1 127° pptd. some crystals, m. 76-7°. The pot residue was 40.8 g. Pure 1,3,4-trichlorobutanol-2 (VII) was obtained by refluxing either the impure chlorohydrin or the ester fraction from the oxdn. of III or IV with 5 times its wt. of N-MeOH-HCl. VII b1 80°, n_{25D} 1.5022.0 With base VII gave an epoxide, b4 96-100°, n_{20D} 1.4749; phenylethan m. 132-3°. Reaction of the chlorohydrin with 20% NaOH gave 80% dichloropropionate, b10 73-5°, n_{20D} 1.4767. The rate of basic hydrolysis of 0.1M chlorohydrin was second order rate. A plot of O consumed against time generally produced typical sigma shaped curves. The following results were obtained (isomer, solvent, temp., concn. of the dichlorobutene, concn. of catalyst, added salt or acid, and the rate + 104 given): IV, AcOH, 90°: 2M, 0.1M, CoOAc, 10%; 1M, 0.1M, CoOAc, 7.6; 2M, 0.05M, CoOAc, 0.015M HCl, 5.5; 2M, 0.05M, CoOAc, 0.2M, LiCl, 3.2; 2M, 0.05M, CoCl₂, 11. 3,4-Dichlorobutene, no solvent, 90°, no catalyst, 1.0, 0.5 mole-% Co as Co naphthenate, 3.2; 15 mole-% K₂CO₃, no reaction. CoOAc, and CoCl₂ represent cobaltous acetate-4H₂O and CoCl₂-6H₂O, resp. III, AcOH, 90°: 2M, 0.1M, CoOAc, 13; 2M, 0.1M, CoOAc, 0.4M, Pb(OAc)₂, 26; 2M, -, CoCl₂, 30-100°: 2M, 0.1M, CoCl₂, 23.80°: 2M, -, CoCl₂, 21; 2M, 0.1M CoCl₂, 1,2-azobisisobutyronitrile, 20.70°: 2M, 0.1M, CoCl₂, 8. III, no solvent, 90°, no catalyst, 10; 0.5 mole-% Co as Co naphthenate, 6.8, no catalyst, 1,2-azobisisobutyronitrile, 10.80°: 0.5% Co as Co naphthenate, 20%; light MgO, 11; no catalyst, 20%; light MgO 13. III, Me₃COH, 90°: 0.4M, negligible reaction.
 ACCESSION NUMBER: 1962:1895 CAPLUS
 DOCUMENT NUMBER: 56:1895
 ORIGINAL REFERENCE NO.: 56:3009-i,301a-i,302a
 TITLE:
 AUTHOR(S): Brill, Wm. F.
 CORPORATE SOURCE: Food Machinery and Chem. Corp., Princeton, NJ
 SOURCE: Journal of Organic Chemistry (1961), 26, 2969-72
 CODEN: JOCEAH; ISSN: 0022-3263
 DOCUMENT TYPE: Journal
 LANGUAGE: Unavailable

L4 ANSWER 9 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN
 AB Epoxides are prepared by treating an ethylenically unsatd. compound with Ach- or Et₂CHO-monopersacrylate, preferably at 40-120°. Thus, 351 g. of a mixture of Ach (75%) and Me₂CO (25%) charged to a cylindrical oxidizer, cooled to -4°, irradiated with ultraviolet light, and O forced through by means of a diffuser gives 46% Ach-monopersacrylate (I) at the end of 2 hrs. Fresh AchMe₂CO may be added at the top of the oxidizer and the oxidized product removed from the bottom. Styrene (413 g.) containing 0.8 g. 4,2,6-C₁₇O₂N₂C₆H₂OH (II) is charged to a still, refluxed at 70° under 60 mm. pressure, 229 g. of a 52% solution of I (prepared as above) added over a period of 80 min. and 10% II in styrene is added simultaneously through the reflux column at a rate of 20 ml./78 min. and the Ach, Me₂CO, and Ach are removed continuously. Distillation of the residual material gives 86 g. styrene oxide, as well as unreacted styrene. Similarly were prepared the following from the corresponding olefins: 4-vinylcyclohexene monoxide, b9 57°; propylene oxide, 9,10-epoxy-4-methyl-2-pentanone, b26 65°; 9,10-epoxysuccinic acid, m. 52°; glycidol, b5 57°; chloroisobutylene oxide, b50 46°; butadiene monoxide; 3,4-epoxycyclohexanecarboxaldehyde, b2.5 76-85°; 9,10-epoxy-stearamide: N-(n-hexyl)-4,5-epoxycyclohexane-1,2-dicarboximide.
 ACCESSION NUMBER: 1956:44861 CAPLUS
 DOCUMENT NUMBER: 50:44861
 ORIGINAL REFERENCE NO.: 50:8730b-e
 TITLE: Epoxidation of ethylenically unsaturated organic compounds
 PATENT ASSIGNEE(S): Union Carbide & Carbon Corp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:
 PATENT NO. KIND DATE APPLICATION NO. DATE
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 GB 735974 ----- 19550831 GB

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN
 AB The object of this work was the preparation and study of the phys. properties
 of certain hydrocarbons with b. ps. in the range of gas oil (petroleum) fraction b. 200-320°. Preparation of hydrocarbons: Aliphatic series: (1) Pentadecane (I). Condensation of (CH₂)₃ with C₆H₁₃MgBr gave 38% heptanol; condensation of C₇H₁₅MgBr with HCO₂Et gave 61% 8-pentadecanol (II), m. 43.5° (from EtOH-H₂O), b11 153° (cf. C.A. 41, 5850e for data on C₆H₁₃Br and C₇H₁₅Br). Dehydration of II in H₂SO₄ gave 7-pentadecene, b20 137°, d20 0.7726, n_{20D} 1.4370, MR (mol. refraction) 71.21, oxidation of which with CrO₃-HOAc yielded heptanoic acid, b. 218-24°, and octanoic acid, b. 233-8°, both characterized by titration and the Ag salts. Catalytic hydrogenation with Pt black of II gave I, b22 145°, d20 0.7638, n_{20D} 1.4286, MR 71.49. (2) 4,6-Dipropynonane could not be prepared. The 4-heptanol (III) prepared in 88% yield from C₃H₇MgBr and HCO₂Et b25 70°, d20 0.8127, n_{20D} 1.4184, MR 36.00; these data compared favorably with those for III prepared from C₃H₇MgBr and C₃H₇CHO, in contrast to the poorer data for III prepared from esters: b25 64°, d20 0.8139, n_{20D} 1.4181, MR 35.92. The C₇H₁₅MgBr was formed with difficulty, EtBr being necessary to prime the reaction, and subsequent condensation with HCO₂Et gave only secondary products, notably 2-propyl-1-pentanol. The preparation of 3-propyl-2-(2-propynyl)hexanoic acid (IV) by malonic ester synthesis (for subsequent decarboxylation to C₁₅H₃₂) was unsuccessful. (3) 2,4,8,10-Tetramethylheptadecane could not be prepared from Me₂CH₂CH₂MgBr and HCO₂Et; only secondary products were obtained. 4-Methyl-2-pentanol, 81% from Me₂CH₂HgBr and Ach, b10 47°, d20 0.80708, n_{20D} 1.4101, MR 31.22; formation of its bromide with P or Br or with HBr was difficult, and subsequent condensation with (CH₂)₃ gave mostly secondary products and 8% 2,4-dimethylpentanol. (4) 2,6-Dimethyl-3,5-disopropylheptane was not formed from (Me₂CH₂)₂CHMgBr and HCO₂Et, but instead (Me₂CH₂)₂CH₂, Me₂CH₂CH₂, and (Me₂CH₂)₂CH₂O₂ were obtained. The malonic ester synthesis was considered improbable after attempted preparation of IV. (5) Hexadecane (V), 60% from 1-bromooctane and Na, b11 153°, d20 0.7770, n_{20D} 1.4351, MR 75.91; in EtOH the reaction is slow, with an intermediate blue color; if 1-bromooctane is added to a suspension of Na in xylene, the initially fast solution of the Na is followed by the slow elimination of Br as NaBr (refluxing 3 h.) (70% yield). (6) 5-Methyl-4,6-dipropynonane. The intermediate (Pr₂CH₂C(OH)Me could not be obtained by Grignard condensation, only secondary products such as 3-propyl-2-hexanol. (7) 4,7-Dipropyldecano (VI). 4-Heptanol (VII), from 4-heptanol and CrO₃-HOAc, b741 142°, d20 0.8116, n_{20D} 1.4065, MR 34.53; semicarbazone m. 135°. BrMgC.tpbond.CMgBr (VIII) was made by passing a stream of pure C₂H₂ into Et₂O and EtMgBr until C₂H₆ evolution ceased and 2 liquid layers appeared; condensation with VII gave 89% acetylenic glycol, m. 120° (from CCl₄), dehydration of which in boiling 25% H₂SO₄ gave 4,7-dipropyl-3,7-decadien-5-yne (IX), b25 142°, d20 0.8111, n_{20D} 1.4862, MR 77.19 (an extraordinary exaltation of 4.04 was noted). Hydrogenation with Pt black gave VI with phys. data different from those found in the literature (cf. C.A. 8, 1579), b23141°, d20 0.7802, n_{20D} 1.4350, MR 75.59. Selective hydrogenation with Raney Ni in EtOH gave instead intermediate products, identified by oxidation: 4,7-dipropyl-3,5,7-decatriene, 4,7-dipropyl-4,6-decadiene, and 4,7-dipropyl-5-decene. (8) 2,4,6,8,10-

(Continued)
 Hexadecane (V), 60% from 1-bromooctane and Na, b11 153°, d20 0.7770, n_{20D} 1.4351, MR 75.91; in EtOH the reaction is slow, with an intermediate blue color; if 1-bromooctane is added to a suspension of Na in xylene, the initially fast solution of the Na is followed by the slow elimination of Br as NaBr (refluxing 3 h.) (70% yield). (6) 5-Methyl-4,6-dipropynonane. The intermediate (Pr₂CH₂C(OH)Me could not be obtained by Grignard condensation, only secondary products such as 3-propyl-2-hexanol. (7) 4,7-Dipropyldecano (VI). 4-Heptanol (VII), from 4-heptanol and CrO₃-HOAc, b741 142°, d20 0.8116, n_{20D} 1.4065, MR 34.53; semicarbazone m. 135°. BrMgC.tpbond.CMgBr (VIII) was made by passing a stream of pure C₂H₂ into Et₂O and EtMgBr until C₂H₆ evolution ceased and 2 liquid layers appeared; condensation with VII gave 89% acetylenic glycol, m. 120° (from CCl₄), dehydration of which in boiling 25% H₂SO₄ gave 4,7-dipropyl-3,7-decadien-5-yne (IX), b25 142°, d20 0.8111, n_{20D} 1.4862, MR 77.19 (an extraordinary exaltation of 4.04 was noted). Hydrogenation with Pt black gave VI with phys. data different from those found in the literature (cf. C.A. 8, 1579), b23141°, d20 0.7802, n_{20D} 1.4350, MR 75.59. Selective hydrogenation with Raney Ni in EtOH gave instead intermediate products, identified by oxidation: 4,7-dipropyl-3,5,7-decatriene, 4,7-dipropyl-4,6-decadiene, and 4,7-dipropyl-5-decene. (8) 2,4,6,8,10-

(Continued)
 1,2-diphenyl-2-propanol, m. 72° (from EtOH), b9 163°, dehydrated in boiling 50% H₂SO₄ to 1,2-diphenyl-2-propene, m. 81° (from EtOH), b22 170°, slightly sol. in Et₂O. Hydrogenation with Pt black was selective, to form XXI, b25 160°, d20 0.9799, n_{20D} 1.5573, MR 64.42. (4) 1,3-Diphenylpropane (XXII). PhCH₂MgCl and HCO₂Et gave only 7-8% 1,3-diphenyl-2-propanol (XXIII), owing to the formation of secondary products such as PhCH₂CH₂OH; the Grignard condensation of PhCH₂CH₂Br with BzH gave 35% 1,3-diphenyl-1-propanol (XXIV), m. 71° (from EtOH), b14 186°. Dehydration of XXII with H₂SO₄ gave trans-1,3-diphenylpropane, m. 51° (from EtOH), b25 159°; similarly XXIV gave a mixt., b15 170°, d20 0.9978, n_{20D} 1.5689, MR 63.68; chilling removed 30% as the solid trans form, and the oily liq. was purified to cis-1,4-diphenylpropane, b18 178°, d20 1.0138, n_{20D} 1.5807, MR 63.75. XXII, formed by hydrogenation with Raney Ni of either isomer, b18 160°, d20 0.9831, n_{20D} 1.5594, MR 64.41. (5) 1,2-Diphenylbutane (XXV), b28 172°, d20 0.9777, n_{20D} 1.5554, MR 68.98. Friedel-Crafts condensation of EtCOCl and C₆H₆ gave Et Ph, b11 97°, d20 1.0103, n_{20D} 1.5256, MR 40.69, which with PhCH₂MgCl gave 1,2-diphenyl-1-butanol, b20 180°, d20 1.0367, n_{20D} 1.5752, MR 72.06. Dehydration in H₂SO₄ or P₂O₅ gave a mixt. of isomeric olefins; subsequent hydrogenation with Raney Ni was selective, to form XXV, attacking only isomers without a double bond between the 2 rings (the formation of XVIII from stilbene is an exception to this general rule). (6) 1,4-Diphenylbutane (XXVI). The 2 diastereoisomers of 1,4-diphenyl-2-butene-1,4-diol, from BzH and VIII, were prep'd. by their different solubilities in Et₂O; on crystn. from EtOH the a-form m. 146° and the B-form m. 99.5°. The products, predominantly u-, resinify easily in acid. Hydrogenation gave the stereoisomeric diols: a-form (XXVII) of 1,4-diphenyl-1,4-butenediol m. 110°; B-form m. 93°. In contrast to the aliph. diols, dehydration of XXVII with 30% H₂SO₄ did not give a diolefin, but instead 1,4-diphenyl-1,4-epoxybutane (2,5-diphenyltetrahydrofuran) (XXVIII), b27 210°, d20 1.0752, n_{20D} 1.5770, MR 63.49, very stable, resistant to H in EtOH with Raney Ni or Pt black, reacts slowly with H and Pt black in HOAc. In contrast, the action of nascent H, from Na and EtOH, is very effective, giving 1,4-diphenyl-1-butanol, b32 218°, d20 1.0347, n_{20D} 1.5573, MR 65.17; dehydration in 60% H₂SO₄ then gave 1,4-diphenyl-1-butene, b14 170°, d20 1.0330, n_{20D} 1.5945, MR 68.37. The XXVI produced by hydrogenation with Raney Ni, m. 52.5°, was identical with that prep'd. from PhCH₂CH₂Br and Na. No rearrangement occurred in the dehydration of XXVII (cf. Tiffeneau, Oskhoff, and Levy, C.A. 26, 2429). (7) 1,2-Diphenylpentane (XXIX), b32 184°, d20 0.9649, n_{20D} 1.5480, MR 73.74. 1,2-Diphenyl-2-pentanol, 94% from PhCH₂MgCl and PbBr₂, b22 184°, d20 0.9913, n_{20D} 1.5882, MR 81.50, was dehydrated with H₂SO₄ to a mixt. (XXX) of isomeric oleins, b27 185°, d20 0.9868, n_{20D} 1.5798, MR 74.85; dehydration with P₂O₅ gave a mixt. (XXXI), b28 196°, d20 0.0026, n_{20D} 1.5807, MR 73.76. XXXI contains mostly 1,2-diphenyl-2-pentene and is smoothly hydrogenated at normal pressure with Raney Ni to XXIX, whereas XXX, contg. mostly 1,2-diphenyl-1-pentene, is not hydrogenated. (8) 2,5-Diphenylhexane (XXIX). 2,5-Diphenyl-3-hexyne-2,5-diol, from VIII and MeBr, was sepd. into the a-form, m. 163.5° (from EtOH), and the predominant B-form (XXXII), m. 126° (from C₆H₆), by the virtual insol. of the u-form in Et₂O. Attempted dehydration caused resin formation, esp. with XXXI and 10% H₂SO₄, when a red resin was obtained. So hydrogenation gave 2,5-diphenyl-2,5-hexanediol as the a-form.

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
 Pentamethylheptadecane. An attempted prepn. by Grignard condensation of Me₂CH₂CH₂CH₂Br with EtOAc gave only secondary products, such as 2,4-dimethyl-1-hexanol, 2,4-dimethyl-1-pentene, and 2,4-dimethylpentane. (9) 2,4,6-Trimethyl-3,5-disopropylheptane could not be prep'd. by Grignard condensation of (Me₂CH₂)₂CHBr (C.A. 41, 5850e) with EtOAc. (10) 2,7-Dimethyl-3,6-disopropyloctane (X). (Me₂CH₂)₂CO, formed by CrO₃ oxidn. of the alc., was condensed with VII to 2,7-dimethyl-3,6-disopropyl-4-octyne-3,6-diol, m. 109° (from CCl₄); the condensation of this ketone, doubly branched in the u-position, goes normally. Dehydration in 20% H₂SO₄ gave 2,7-dimethyl-3,6-disopropyl-2,6-octadien-4-yn (XI), b28 142°, d20 0.8360, n_{20D} 1.5050, MR 77.34. Catalytic hydrogenation with Raney Ni at 180° and 120 kg./sq. cm. gave X, b35 142°, d20 0.7960, n_{20D} 1.4470, mol. refraction 75.78; hydrogenation with Pt black gave instead 2,7-dimethyl-3,6-disopropyl-4-octene (XII), b21 132°, d20 0.8157, n_{20D} 1.4513, MR 73.99; hydrogenation with Raney Ni (room temp. and 1 atm.) gave intermediate products, 2,7-dimethyl-3,6-disopropyl-2,4,6-octatriene, and 2,7-dimethyl-3,6-disopropyl-3,5-octadiene, as well as XI. (11) 5,9-Dibutyldecane (XIII). Bu₃MgBr and HCO₂Et gave 92% 5-nonenol, b27 103°, d20 0.8257, n_{20D} 1.4289, MR 44.95, oxidized with CrO₃ to 5-nonanone; condensation with VII gave 83% 5,8-dibutyl-6-dodecayne, 5,8-diol, m. 130.5° (from EtOH). Dehydration in 25% H₂SO₄ gave 5,8-dibutyl-4,8-dodecadien-6-yn (XIV), b29 189°, d20 0.8222, n_{20D} 1.4866, MR 95.77; total hydrogenation with Pt black in HOAc yielded XIII, b32 192°, d20 0.7922, n_{20D} 1.4436, MR 94.48. Hydrogenation with Raney Ni in EtOH gave intermediate compds. (12) 2,9-Dimethyl-4,7-disobutyldecane (XV), prep'd. similarly to XIII in 81% yield, b30 167°, d20 0.7882, n_{20D} 1.4412, MR 94.52. 2,9-Dimethyl-4,7-disobutyl-5-decayne-4,7-diol, m. 72° (from ligroin), was dehydrated in 25% H₂SO₄ to 2,9-dimethyl-4,7-disobutyl-3,7-decadien-5-yne (XVI), b28 156°, d20 0.8023, n_{20D} 1.4783, MR 95.94. Total hydrogenation to XV was possibly only at a pressure of 130 kg./sq. cm. at 180° with Raney Ni. (13) The Wurtz reaction gave very poor yields of the desired hydrocarbons; the main products were dimers and "intermol." products of free radicals. Condensation of 1-bromodecan and iso-AMBr in Bu₂Na with Na gave a small quantity of impure 2-methyltetradecane, b22 152°, d20 0.7832, n_{20D} 1.4290, MR 71.75; the main products were Me₂CH₂Et, the 2-methylbutenes, and the dimer of the decyl radical, eicosane. Instead of 2methyl-4-isobutyldecane from 1-bromohexane and (Me₂CH₂)₂CHBr, only secondary products such as dodecane were formed. Tetradecane (XVII), and not the desired 2-methyl-4-isobutylheptadecane, was the main product from 1-bromohexane and (Me₂CH₂)₂CHBr, b23 135°, d20 0.7700, n_{20D} 1.4303, MR 66.46. PrCH₂CH₂CH₂Me and Na in xylene gave only "intermol." rearrangement products and not the desired dimer, 2,7-dimethyl-4,6-disopropyloctane. Arylaliph. series: (1) 1,2-Diphenylethane, from PhCH₂MgCl and BzH, m. 69° (from CCl₄); dehydration in 50% H₂SO₄ gave stilbene, m. 124° (from EtOH), and hydrogenation with Raney Ni easily formed XVIII, m. 52°. (2) 1,1-Diphenylpropane (XIX). 1,1-Diphenyl-1-propanol (XX), in 87% yield from EtMgBr and PbBr₂, m. 96° (from 90% EtOH) [cf. Klages, Ber. 35, 2647 (1902)], was dehydrated by distn. in vacuo with activated clay to 1,1-diphenyl-1-propene, b15 154°, d20 1.0250, n_{20D} 1.5880, MR 63.70; hydrogenation with Raney Ni gave XIX, b18 145°, d20 0.9975, n_{20D} 1.5701, MR 64.48. Dehydration of XX in boiling EtOH easily gave the dimer 2,3-dimethyl-1,1,4-tetraphenyl-1-butene, m. 212°; the structure was detd. by Br no., mol. wt. by f.p. in C₆H₆, and oxidn. (3) 1,2-Diphenylpropane (XXI). PhCH₂MgCl and AcPh gave 92%

(Continued)
 L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
 1,2-diphenyl-2-propanol, m. 72° (from EtOH), b9 163°, dehydrated in boiling 50% H₂SO₄ to 1,2-diphenyl-2-propene, m. 81° (from EtOH), b22 170°, slightly sol. in Et₂O. Hydrogenation with Pt black was selective, to form XXI, b25 160°, d20 0.9799, n_{20D} 1.5573, MR 64.42. (4) 1,3-Diphenylpropane (XXII). PhCH₂MgCl and HCO₂Et gave only 7-8% 1,3-diphenyl-2-propanol (XXIII), owing to the formation of secondary products such as PhCH₂CH₂OH; the Grignard condensation of PhCH₂CH₂Br with BzH gave 35% 1,3-diphenyl-1-propanol (XXIV), m. 71° (from EtOH), b14 186°. Dehydration of XXII with H₂SO₄ gave trans-1,3-diphenylpropane, m. 51° (from EtOH), b25 159°; similarly XXIV gave a mixt., b15 170°, d20 0.9978, n_{20D} 1.5689, MR 63.68; chilling removed 30% as the solid trans form, and the oily liq. was purified to cis-1,4-diphenylpropane, b18 178°, d20 1.0138, n_{20D} 1.5807, MR 63.75. XXII, formed by hydrogenation with Raney Ni of either isomer, b18 160°, d20 0.9831, n_{20D} 1.5594, MR 64.41. (5) 1,2-Diphenylbutane (XXV), b28 172°, d20 0.9777, n_{20D} 1.5554, MR 68.98. Friedel-Crafts condensation of EtCOCl and C₆H₆ gave Et Ph, b11 97°, d20 1.0103, n_{20D} 1.5256, MR 40.69, which with PhCH₂MgCl gave 1,2-diphenyl-1-butanol, b20 180°, d20 1.0367, n_{20D} 1.5752, MR 72.06. Dehydration in H₂SO₄ or P₂O₅ gave a mixt. of isomeric olefins; subsequent hydrogenation with Raney Ni was selective, to form XXV, attacking only isomers without a double bond between the 2 rings (the formation of XVIII from stilbene is an exception to this general rule). (6) 1,4-Diphenylbutane (XXVI). The 2 diastereoisomers of 1,4-diphenyl-2-butene-1,4-diol, from BzH and VIII, were prep'd. by their different solubilities in Et₂O; on crystn. from EtOH the a-form m. 146° and the B-form m. 99.5°. The products, predominantly u-, resinify easily in acid. Hydrogenation gave the stereoisomeric diols: a-form (XXVII) of 1,4-diphenyl-1,4-butenediol m. 110°; B-form m. 93°. In contrast to the aliph. diols, dehydration of XXVII with 30% H₂SO₄ did not give a diolefin, but instead 1,4-diphenyl-1,4-epoxybutane (2,5-diphenyltetrahydrofuran) (XXVIII), b27 210°, d20 1.0752, n_{20D} 1.5770, MR 63.49, very stable, resistant to H in EtOH with Raney Ni or Pt black, reacts slowly with H and Pt black in HOAc. In contrast, the action of nascent H, from Na and EtOH, is very effective, giving 1,4-diphenyl-1-butanol, b32 218°, d20 1.0347, n_{20D} 1.5573, MR 65.17; dehydration in 60% H₂SO₄ then gave 1,4-diphenyl-1-butene, b14 170°, d20 1.0330, n_{20D} 1.5945, MR 68.37. The XXVI produced by hydrogenation with Raney Ni, m. 52.5°, was identical with that prep'd. from PhCH₂CH₂Br and Na. No rearrangement occurred in the dehydration of XXVII (cf. Tiffeneau, Oskhoff, and Levy, C.A. 26, 2429). (7) 1,2-Diphenylpentane (XXIX), b32 184°, d20 0.9649, n_{20D} 1.5480, MR 73.74. 1,2-Diphenyl-2-pentanol, 94% from PhCH₂MgCl and PbBr₂, b22 184°, d20 0.9913, n_{20D} 1.5882, MR 81.50, was dehydrated with H₂SO₄ to a mixt. (XXX) of isomeric oleins, b27 185°, d20 0.9868, n_{20D} 1.5798, MR 74.85; dehydration with P₂O₅ gave a mixt. (XXXI), b28 196°, d20 0.0026, n_{20D} 1.5807, MR 73.76. XXXI contains mostly 1,2-diphenyl-2-pentene and is smoothly hydrogenated at normal pressure with Raney Ni to XXIX, whereas XXX, contg. mostly 1,2-diphenyl-1-pentene, is not hydrogenated. (8) 2,5-Diphenylhexane (XXIX). 2,5-Diphenyl-3-hexyne-2,5-diol, from VIII and MeBr, was sepd. into the a-form, m. 163.5° (from EtOH), and the predominant B-form (XXXII), m. 126° (from C₆H₆), by the virtual insol. of the u-form in Et₂O. Attempted dehydration caused resin formation, esp. with XXXI and 10% H₂SO₄, when a red resin was obtained. So hydrogenation gave 2,5-diphenyl-2,5-hexanediol as the a-form.

with Na: only C₅H₁₁, C₅H₁₀, C₁₀H₂₂, and dimethylolactane were formed. The condensation of C₅H₁₁MgBr with p-Bz₂C₆H₄ with FeCl₃ (cf. C.A. 39, 2739, 6) was also unsuccessful. (3) 1,2-Dicyclohexylmethane (XL), obtained by the hydrogenation at 180° with Raney Ni of PhCH₂CH₂OH, b19 140°, d20 0.8825, n_{20D} 1.4802, MR 62.47. (4) 1,1-Dicyclohexylpropane (XLII), from Ph₂CH₂CH₂OH by hydrogenation with Raney Ni, b14 131°, d20 0.9023, n_{20D} 1.4935, MR 67.05. Condensation of EtCO₂Et and C₆H₁₁MgCl gave only secondary products: C₆H₁₂, C₆H₁₁CH₂Ac, and cyclohexylpropanol. (5) 1,2-Dicyclohexylpropane (XLII), from the hydrogenation of PhCH₂CH₂OH with Pt black, b15 149°, d20 0.8819, n_{20D} 1.4791, MR 66.90. (6) 1,3-Dicyclohexylpropane (XLIII), from the hydrogenation of PhCH₂CH₂OH in HOAc with Pt black, b24 151°, d20 0.8740, n_{20D} 1.4750, MR 67.01; the hydrogenation was nonselective, acting equally on the olefinic bond and the arom. nuclei. (7) 1,2-Dicyclohexylbutane (XLIV), obtained from PhCH₂CH₂OH with Pt by hydrogenation with Raney Ni at 185°, b22 160°, d20 0.9146, n_{20D} 1.5038, MR 71.85. (8) 1,4-Dicyclohexylbutane (XLV), obtained similarly from [CH₂CH₂CH₂OH]₂ at 180°, b19 167°, d20 0.8731, n_{20D} 1.4750, MR 71.69. (9) 1,2-Dicyclohexylpentane (XLVI), prep'd. similarly from PhCH₂CH₂OH, b17 157°, d20 0.9218, n_{20D} 1.5092, MR 76.47. (10) 2,5-Dicyclohexylhexylbenzene (XLVII), from [CH₂CH₂CH₂OH]₂, b32 201°, d20 0.8901, n_{20D} 1.4876, MR 80.87. (11) 1,1,2-Triphenylmethane was hydrogenated to 1,1,2-tricyclohexylmethane (XLVIII), b30 230°, d20

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
1.0236, n_{20D} 1.5786, MR 90.21. Studies of hydrocarbon properties as a function of mol. structure: Generally the aliph. hydrocarbons show increasing d. and n with increase in branching; the b. ps. decrease. The polyolefinic hydrocarbons show a trend in d. and n with the C/H ratio or degree of unsatn. The mol. exaltation due to the acetylenic group is roughly 3 times that for ethylenic bonds. The following phys. data (see also under each of the preceding individual syntheses), i.e., the mol. exaltation (obsd. mol. refraction - calcd. value), the kinematic

viscosity

in centistokes, and the abs. viscosity in centipoises, are given: XVII, -0.42, 3.45, 2.66; I, -0.01, 3.75, 2.35; V, -0.21, 4.33, 3.37; VI, -0.53, 3.71, 2.90; IX, -0.34, 4.65, 3.71; XI, -0.12, 10.28, 8.14; XII, -0.08, 9.64, 7.60; for the following only the mol. exaltation is given: IX, +4.04; XI, +4.19; XIV, +4.11; 5,8-dibutyl-4,6,8-dodecatriene, +2.02; 5,8-dibutyl-5,7-dodecadiene, +1.01; XVI, +4.28; 2,9-dimethyl-4,7-diisobutyl-3,5,7-decatriene, +2.16; 2,9-dimethyl-4,7-diisobutyl-4,6-decadiene, +1.02. The phys. state of the arylaliph. hydrocarbons is intimately bound to the mol. structure esp. the degree of symmetry.

Asym.

compds. are liq. whereas the sym. ones are solid. The d. and n of liqs. decrease with increasing chain length (or decrease in C/H ratio) and with increasing degree of symmetry. Phys. data (read as above): XIX, +0.18, 5.07, 5.06; XXI, +0.12, 6.74, 6.61; XXII, +0.15, 7.81, 7.83; XXV, +0.06, 9.36, 9.15; XXIX, +0.20, 12.00, 11.58. With the cyclic hydrocarbons, the d. and n of the sym. isomers are lower than of the asym. ones. Phys.

data

(read as above): XXXIX, +0.05, 6.14, 5.01; XL, -0.01, 7.90, 6.96; XLI, -0.05, 9.68, 8.74; XLII, -0.20, 11.52, 10.16; XLIII, -0.09, 8.91, 7.80; XLIV, +0.13, 10.99, 10.05; XLV, -0.03, 12.71, 11.17; XLVI, +0.13, 14.68, 13.53; XLVII, -0.09, 23.35, 20.79; XLVIII, 0.00, 122.85, 127.80; XXXVI,

m.

52.5°, b₁₆ 166°; XXXVII, b₁₆ 172°, -0.03, 41.56, 41.40. The viscosity of the 3 series above at 20° increases with the length of C chain, but not with the C/H ratio; the arylaliph. asym. hydrocarbons have lower values than the corresponding cyclic compds. An increase occurs with the appearance of closed rings, also with degree of symmetry for compds. of analogous structure. More evidence is needed to confirm these generalities. Conclusions: (1) The Wurtz reaction does not go so simply as given in the std. texts; free radicals play an important part. Temp. is important, esp. in the isomerization of the radicals, and it is difficult to isolate pure products, due to secondary products. (2) The Grignard condensation is a better method for obtaining the desired structures, with min. rearrangement of the reactants. (3) Dehydration

and

dehydrogenation depend greatly on mol. structure; the acetylenic diols dehydrate differently in the aliph. and arylaliph. series. After removal of the triple bond, epoxides are formed. Compds. of the type ArCH₂CRAr are resistant to catalytic hydrogenation, so that selective redn. to arylaliph. or to cyclic hydrocarbons can be accomplished. Similarly, acetylenic diols are also selectively hydrogenated; the

partial

redn. of the triple bond is characterized by (1) a slower reaction, (2) change from exothermic to endothermic reaction, and (3) pptn. of the insol. olefin from EtOH. The hydrogenation of the sym. diaryl 1,4-epoxides is free of intramol. rearrangement.

ACCESSION NUMBER: 1948:27383 CAPLUS

DOCUMENT NUMBER: 42:27383

ORIGINAL REFERENCE NO.: 42:5833f-i, 5834e-i, 5835a-i, 5836a-i, 5837a-i, 5838a-e

TITLE: Aliphatic, arylaliphatic, and cyclic (C14-C20) hydrocarbons. Synthesis

L4 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2007 ACS on STN (Continued)
AUTHOR(S): Tuot, Marcel; Guyard, Marcelle
CORPORATE SOURCE: Ecole natl. supérieure pétrole, Strasbourg
SOURCE: Bulletin de la Société Chimique de France (1947) 1086-96
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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| => FIL STNGUIDE | SINCE FILE | TOTAL |
| COST IN U.S. DOLLARS | ENTRY | SESSION |
| FULL ESTIMATED COST | 57.80 | 58.01 |
| DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) | SINCE FILE | TOTAL |
| CA SUBSCRIBER PRICE | ENTRY | SESSION |
| | -7.80 | -7.80 |

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FILE 'CAPLUS' ENTERED AT 11:23:22 ON 28 AUG 2007

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|----|---|
| L1 | 4941 S OLEFIN AND DISTILLATION |
| L2 | 14686 S PROPANE AND (PROPENE OR PROPYLENE) |
| L3 | 207 S L1 AND PROPANE AND (PROPENE OR PROPYLENE) |
| L4 | 10 S L3 AND (EPOXIDATION OR EPOXIDE) |

FILE 'STNGUIDE' ENTERED AT 11:35:30 ON 28 AUG 2007

=> s l3 and (compress or compressed)

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|----|-----------------------------------|
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| 0 | DISTILLATION |
| 0 | PROPANE |
| 0 | PROPENE |
| 0 | PROPYLENE |
| 0 | COMPRESS |
| 89 | COMPRESSED |
| L5 | 0 L3 AND (COMPRESS OR COMPRESSED) |

=> s l1 and (compress or compressed)

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| 0 | OLEFIN |
| 0 | DISTILLATION |
| 0 | COMPRESS |
| 89 | COMPRESSED |
| L6 | 0 L1 AND (COMPRESS OR COMPRESSED) |

=> s l1

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|----|---------------------------|
| 0 | OLEFIN |
| 0 | DISTILLATION |
| L7 | 0 OLEFIN AND DISTILLATION |

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| => file caplus | SINCE FILE | TOTAL |
| COST IN U.S. DOLLARS | ENTRY | SESSION |
| FULL ESTIMATED COST | 1.44 | 59.45 |
| DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) | SINCE FILE | TOTAL |
| CA SUBSCRIBER PRICE | ENTRY | SESSION |
| | 0.00 | -7.80 |

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      2128 COMPRESSES
      5734 COMPRESS
          (COMPRESS OR COMPRESSES)
      67481 COMPRESSED
L8          8 L3 AND (COMPRESS OR COMPRESSED)

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L8 ANSWER 1 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
 AB A low-pressure olefin recovery process and plant are described in which feed gas is compressed and distilled at a primary distillation pressure. The overhead stream is chilled at <30 kg/cm² to partially condense the overheads. The primary distillation tower is refluxed with at least a portion of the condensate. The overhead vapor is further chilled and partially condensed and the condensate is fed to a demechanizer. The remaining vapor is cooled in a cold section and the resultant liquid is phase separated and expanded to provide refrigeration for the cold section. The expanded vapor from the cold section is recycled to the process gas compressor. The bottoms streams from the primary distillation zone and the demechanizer are fractionated into resp. streams consisting essentially of ethylene, ethane, propylene, propane, C4's, and C5+. Process flow diagrams are presented.

ACCESSION NUMBER: 2006:9427 CAPLUS
 DOCUMENT NUMBER: 144:88707
 TITLE: Low-pressure olefin recovery process
 INVENTOR(S): Verma, Vijender K.; Hu, Jichuan
 PATENT ASSIGNEE(S): Kellogg Brown & Root, Inc., USA
 SOURCE: U.S. Pat. Appl. Publ., 19 pp.
 CODEN: USXXCO
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
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| US 2006004242 | A1 | 20060105 | US 2004-884659 | 20040702 |
| CA 2497943 | A1 | 20060102 | CA 2005-2497943 | 20050222 |
| IN 2005K000152 | A | 20070202 | IN 2005-K0152 | 20050310 |
| EP 1637577 | A2 | 20060322 | EP 2005-5372 | 20050311 |
| R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, BA, HR, IS, YU | | | | |
| CN 1715260 | A | 20060104 | CN 2005-10082196 | 20050701 |
| MX 2005PA07272 | A | 20060111 | MX 2005-PA7272 | 20050701 |
| PRIORITY APPLN. INFO.: | | | US 2004-884659 | A 20040702 |

L8 ANSWER 2 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
 AB Ethylene (I) and propylene (II) are separated from the raw gas, formed from pyrolysis of hydrocarbons, by washing the raw gas at 8-12 atmospheric with an N-alkyl-γ or 8-lactam to dissolve acetylenes, diolefins, H2S, S-containing organic compds. and C4 hydrocarbons. The washed gas contains I, II, H2S, CO, H, CH4, CO2, ethane, propane, and butane. Preferred lactam for washing is N-methylpyrrolidinone (III). The washed gas is treated with an aqueous alkali solution to remove traces of H2S and CO2. The purified gas is compressed to 25-35 atmospheric, and either condensed by cooling it at very low temperature, or absorbed by a liquid. The condensate or the solution is distilled to give pure I and II. The components absorbed by III, namely, acetylene, diolefins, H2S, S-containing organic compds. and a part of CO2, are regenerated by evaporation. The regenerating column is a continuous distillation column, fed with the washing liquor and with steam at such a rate that at the head of the column a part of water and the regenerated components are distilled. The III in the residue contains 1-10% water. The hot residual III is dehydrated by bubbling through it a part of CH4 and of H, released in the purification. Acetylene and diolefins are partly polymerized during the regeneration. The consumption of alkali is low. A design of the plant is given.

ACCESSION NUMBER: 1966:419864 CAPLUS
 DOCUMENT NUMBER: 65:19864
 ORIGINAL REFERENCE NO.: 65:36495-h
 TITLE: Separation of ethylene and propylene from raw pyrolyzed gas
 INVENTOR(S): Thormann, Kurt
 PATENT ASSIGNEE(S): Metallgesellschaft A.-G.
 SOURCE: 5 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|------|----------|-----------------|----------|
| DE 1183902 | | 19641223 | DE 1960-M45718 | 19600623 |
| PRIORITY APPLN. INFO.: | | | DE | 19600623 |

L8 ANSWER 3 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
 AB The process of U.S. 2,-600,110 (CA 46, 8358h), which is based on the heat-pump principle, is improved by passing a major portion of the compressed vapors of the kettle product refrigerant to the kettle section of the fractionator and compressing the rest of the vapors in a 2nd compression step so that they are condensed by available cooling water, thus increasing the efficiency of heat transfer. The improved process is particularly adapted to the separation of C2H4 from C2H6, C3H6 from C3H8, and C4H8 from C4H10.
 ACCESSION NUMBER: 1966:57911 CAPLUS
 DOCUMENT NUMBER: 64:57911
 ORIGINAL REFERENCE NO.: 64:10793d-e
 TITLE: Fractional distillation
 INVENTOR(S): Palen, Joseph W.; Moon, John J.
 PATENT ASSIGNEE(S): Phillips Petroleum Co.
 SOURCE: 5 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|------|----------|-----------------|----------|
| US 3229471 | | 19660118 | US 1961-160066 | 19611218 |
| PRIORITY APPLN. INFO.: | | | US | 19611218 |

L8 ANSWER 4 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
 AB Oxidation products, e.g. ethylene oxide, acrolein, and methacrolein, are prepared by the controlled catalytic oxidation of corresponding olefinic hydrocarbons with O₂. For example, the reaction mixture obtained by the catalytic oxidation of propylene with O₂ in the vapor phase in the presence of a Cu2O catalyst and quenching with H₂O contains essentially 2.6% acrolein, 34.2% propylene and propane, 0.8% O₂, 19.4% permanent gases, and 43% water. The mixture is cooled from 149° to 49° in 2 stages. Two separate liquid condensates are obtained, the first containing 8.8% acrolein and 76% water and the second containing 4.2% acrolein and 13% water. The remaining vapor phase contains 97% acrolein, 1.3% water, the hydrocarbons that are gaseous under normal conditions, and the permanent gases of the reaction mixture. The vapor phase is compressed from its pressure of 5.6 atmospheric to 21 atmospheric and is introduced into an absorption tower at the bottom. A jet of water is introduced at the top. The first liquid condensate, separated during the quenching of the reaction mixture, is introduced into the absorption tower at a place midway between the water inlet and the vapor phase inlet. The absorption tower is maintained at 21° and 21 atmospheric. Acrolein is selectively absorbed by the aqueous solvent, forming a solution of water and acrolein in a proportion of 50.1 moles/water/mole acrolein. The gases leaving at the top of the tower contain essentially propylene, propane, and permanent gases. The absorption solution is withdrawn from the bottom of the tower and is distilled with the second condensate. The top fraction from the distillation contains acrolein and a relatively small amount of water. More than 99% acrolein is recovered from the reaction mixture. The aqueous phase at the bottom of the column is partially returned to the absorption tower. A total of 17.6 moles water are introduced at the top of the tower per mole of acrolein which is recovered by distillation. If this reaction is conducted under essentially the same conditions, with the exception that the vapor phase is introduced into the absorption tower without previous compression and both liquid condensates are distilled directly in the distillation column, 35.2 moles water are required per mole acrolein to obtain an equivalent yield of acrolein.

ACCESSION NUMBER: 1964:16023 CAPLUS
 DOCUMENT NUMBER: 60:16023
 ORIGINAL REFERENCE NO.: 60:2772a-e
 TITLE: Recovery of water-soluble oxidation products of hydrocarbons
 INVENTOR(S): Courter, Martin L.; Thayer, David S.
 PATENT ASSIGNEE(S): Shell Internationale Research Maatschappij N. V.
 SOURCE: 6 pp.
 DOCUMENT TYPE: Patent
 LANGUAGE: Unavailable
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------|------|----------|-----------------|----------|
| DE 1147932 | | 19630502 | DE 1961-873353 | 19610406 |
| US 3097215 | | 19630709 | US 1960-21003 | 19600408 |

L8 ANSWER 5 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
AB Chilling of alkylation mixts. rich in isobutane is accomplished by flash vaporization or "self refrigeration." Vapors rich in isobutane are then compressed and fed to high-pressure fractionating towers and isobutane-rich liquids returned to the reactors. The temperature of the system

under the proposed conditions is maintained lower than usual and decomposition

of acidic components in the alkylate is reduced during deisobutanization. The isobutane and olefinic material are treated with a catalyst in the liquid phase at <100°F. With a H2SO4 catalyst, alkylation temps. of 30-55°F. are preferred. The alkylatable material is usually an olefinic hydrocarbon, e.g. propylene or butylene, or it may be an alkyl ester. For example, 30.2 bbl. propylene, 19.2 bbl. butylene, and 539.8 bbl. isobutane were treated in the liquid phase at 50 lb./in.2 gage with 92% H2SO4. The reaction mixture was cooled by

indirect heat-exchange with flashed effluent hydrocarbons, separated, and deisobutanized.

ACCESSION NUMBER: 1963:440793 CAPLUS

DOCUMENT NUMBER: 59:40793

ORIGINAL REFERENCE NO.: 59:7292f-g

TITLE: Alkylation of olefinic hydrocarbons

INVENTOR(S): Davies, James A.

PATENT ASSIGNEE(S): Texaco Inc.

SOURCE: 4 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|-------|----------|-----------------|----------|
| ----- | ---- | ----- | ----- | ----- |
| US 3097250 | ----- | 19630709 | US 1960-39370 | 19600628 |
| PRIORITY APPLN. INFO.: | ----- | ----- | US | 19600628 |

L8 ANSWER 6 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
AB A normally gaseous hydrocarbon component is separated from a mixture of normally gaseous hydrocarbons by introducing the mixture into a distillation zone. A bottom fraction and a vaporous overhead fraction are withdrawn from the distillation zone. A portion of the overhead fraction, which comprises the component which is to be separated, is compressed and thereby heated. A portion of the compressed overhead fraction, which has been further heated or cooled, is brought into contact indirectly with liquid from the lower portion of the distillation zone thereby heating and partially vaporizing the liquid from the distillation zone and partially condensing the overhead. The condensed overhead may be used to reflux the distillation zone, while the vaporized liquid may be used to reboil the distillation zone. This process may be used in the separation of C2H4 from C2H6 in a C2-splitting operation or for the separation of C3H6 from C3H8.

ACCESSION NUMBER: 1962:455562 CAPLUS

DOCUMENT NUMBER: 57:55562

ORIGINAL REFERENCE NO.: 57:10979a-c

TITLE: Gas separation, especially in production of ethylene and propylene

INVENTOR(S): Greco, Saverio G.

PATENT ASSIGNEE(S): M. W. Kellogg Co.

SOURCE: 6 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|-------|----------|-----------------|----------|
| ----- | ---- | ----- | ----- | ----- |
| US 3000188 | ----- | 19610919 | US 1956-622291 | 19561115 |
| PRIORITY APPLN. INFO.: | ----- | ----- | US | 19561115 |

L8 ANSWER 7 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
AB Isobutane (60-90 volume % of the hydrocarbon charge) and olefins, such as propene, react in the liquid phase in the presence of a nonvolatile alkylation catalyst, e.g. H2SO4 (88-94 weight % titratable acidity). The temperature and pressure conditions (30-75°F. and 0-30 lb./sq. in. gage are such that part of the isobutane and practically all of the C3H8 and lighter products are evaporated to give effective refrigeration for the alkylation zone. The alkylator delivers a depropanized liquid emulsion to be settled and caustic-washed free of acid. This alkylate passes to the top of a distillation tower where butanes are distilled from the downcoming alkylate and returned to the alkylator. The isobutane-rich vapors from the alkylator containing C3H8 are compressed (40-130 lb./sq. in. gage) and condensed with cooling water. The condensate is depropanized, and the isobutane-rich bottoms are

returned to the alkylator.

ACCESSION NUMBER: 1962:443938 CAPLUS

DOCUMENT NUMBER: 57:43938

ORIGINAL REFERENCE NO.: 57:8811f-h

TITLE: Catalytic alkylation of olefins with isobutane

INVENTOR(S): Texaco Development Corp.

SOURCE: 9 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------------------|-------|----------|-----------------|----------|
| ----- | ---- | ----- | ----- | ----- |
| GB 895178 | ----- | 19620502 | GB 1960-15908 | 19600505 |
| PRIORITY APPLN. INFO.: | ----- | ----- | US | 19590511 |

L8 ANSWER 8 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN
AB The liquid mixture of hydrocarbons containing olefins, e. g.,
propane and propylene, is freed of these components by
successive decantations in a specially designed apparatus. There the
olefinic
components and the esterifying solution (a 75% H₂SO₄ solution) are
allowed to
circulate in countercurrent manner by means of flowing in an inert gas
preliminarily compressed. The temperature in the battery is
40° and the pressure 20 kg. per sq. cm. At one end of the battery
is collected a liquid containing saturated hydrocarbons and practically
free of
olefins, and at the other an esterifying solution saturated with
olefin alcs. which is caused to appear there by dilution and heating.
The liberated alcs. are isolated by suitable means, e. g., distn
. or solvent extraction. Drawing of apparatus is included.

ACCESSION NUMBER: 1944:25045 CAPLUS

DOCUMENT NUMBER: 38:25045

ORIGINAL REFERENCE NO.: 38:3664i,3665a-b

TITLE: Esterification of olefins

INVENTOR(S): Guinot, Henri M.

PATENT ASSIGNEE(S): Alien Property Custodian.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

| PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|------------|------|----------|-----------------|------|
| US 2345114 | | 19440328 | US | |

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| | 0.00 | -14.04 |

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